

# Effect of Thermo-Plastic Treatment on Structure and Corrosion Properties of High Nitrogen Cr-Steels

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## Abstract

Effect of aging on structure and corrosion properties in high strength austenitic Cr-steels alloying with nitrogen Cr21N1 and Cr18Ni2N1 has been investigated. Structure of the steels and kinetics of austenite decomposition during the treatment was studied by X-ray diffraction, TEM-analyses and electric resistance measurements. The highest values of strength, micro hardness, corrosion rate and relative strength reduction as far as brittle intergranular fracture were observed at the early stage of austenite decomposition at the aging. The formation of nitrides CrN as well martensite stress induced took place in the steels. According to resistometer investigations of nitrogen steels in compare with carbon steel Cr18C1 at the early stage of aging (350 °C) the metastable ordered clusters enriched with chromium and nitrogen were formed. Growths of aging temperature up to 500 °C led to dissolve the carbides or nitrides but at 600 °C discontinuous precipitations were observed nearby the grain boundaries.

Effect of aging combined with cold plastic deformation (CPD) at an 8-24 % reduction by two schemes, before and after aging at 350 °C, on structure and related corrosion properties of steel Cr18Ni2N1 has been studied. CPD at an 8-12 % reduction before aging at 350 °C was shown to thrice decreasing of corrosion rate of the steel as compared to the undeformed state. A thermoplastic treatment by the second scheme with using CPD at an 20% reduction after aging at 350 °C is found to be more effective: the corrosion rate decreased by an order and strength reduction decreased more than by fourfold as compared to the undeformed state.

## Keywords

*Austenite Decomposition; Ordered Clusters; Stress-Martensite; Lattice Parameter; Stress Corrosion Cracking; Cold Plastic Deformation.*

## Introduction

High nitrogen steels (HNS) due to their more strength and corrosion resistant than in other austenitic steels

with carbon are widely used in power machine building, shipbuilding and other fields of engineering. In the nearest future [1-9] after perfecting their manufacturing technology they are expected will be applied in a bigger industrial scale. One of the main directions of investigations to solve these problems is searching a way to increase their resistance to stress corrosion cracking (SCC) both by efficient alloying and optimization of strengthening procedure.

Well known that nitrogen includes a significant contribution in hardening of solid solution and grain boundaries as well as in strain and precipitation hardening. According to data from work [2], realization of these factors at appropriate degree of cold plastic deformation (CPD) can provide extremely high strength for this class of steels up to 3600 MPa. However in a certain structural state they are subjected to SCC in corrosive environment [10, 11] so in this case expedient to use them after strengthening not more then up to  $\sigma_{0.2}=1400$  MPa.

## Materials and Procedure

The high-strength low-carbon steels with high nitrogen content (0.9-1.0%) Cr21N1 and Cr18Ni2N1 have been investigated to study mechanism and kinetics of austenite decomposition and clearing up CPD influence on structure and corrosion properties of steels. Kinetics of decomposition of the oversaturated  $\gamma$ -solid solution in HNS was carried out as compared with high carbon steel Cr18C1. Their chemical composition is presented in Table 1, first two of them were produced by nitrogen pressure casting, homogenization at 1250 °C and rolling with further quenching in water after being exposed for one and half an hour at 1200 °C. The high carbon steel was

manufactured by standard technology and quenched from the same temperature in oil to put the austenite structure. The specimens prepared for testing were aged at 300-500 °C for two hours and at 600 °C for an hour.

TABLE I CHEMICAL COMPOSITION OF STEELS

Steel grade	Content in the steel, wt %							
	C	N	Mn	Si	P	S	Cr	Ni
Cr21N1	0.020	1.021	0.19	0.42	0.015	0.010	21.45	-
Cr18Ni2N1	0.008	0.899	0.30	0.27	0.020	0.010	17.86	2.00
Cr18C1	0.920	-	0.63	0.79	0.025	0.023	18.30	-

The general corrosion and SCC tests were performed in a 3.5% NaCl aqueous solution as it described in the work [10]. Fractographic studies of fracture surface after SCC were examined on a JSM-35C scanning electron microscope and metallographic examination – on a Neophot-32 optical microscope.

The electric resistance measurements were carried out to investigate the kinetics of solid solution decomposition [12]. X-ray diffraction analysis was carried out on a DRON-3 diffractometer with cobalt radiation and a  $\beta$ -radiation filter. The austenite and martensite contents in the steels were estimated using the method of homologous pairs by comparing the integrated intensities of the  $(110)_\alpha$  and  $(111)_\gamma$  lines [13].

The structure of austenite and the type, morphology, and size of additional phases were investigated by transmission electron microscopy on an EMV-100L electron microscope.

## Results and Discussion

Mechanical properties of high nitrogen steels were following: yield strength ( $\sigma_{0.2}$ ) is 650 and 610 MPa; specific elongation ( $\delta$ ) is 21 and 18% after quenching and  $\sigma_{0.2}$ =780 and 820 MPa;  $\delta$ =5 and 9% in aged state respectively in Cr21N1 and Cr18Ni2N1 steels. The highest growth of strength, corrosion rate and susceptibility to SCC in 3.5%-NaCl was observed after aging at 350 °C [14]. The fracture of specimens was brittle and intergranular in this case (Fig. 1, a) while the destruction was not associated with nitrides, located along the grain boundaries. After over-aging the resistance to SCC increased and the intergranular fracture was replaced to quasi-cleavage including

related to colonies of discontinuous decomposition of  $\gamma$ -solid solution (Fig. 1, b). The investigated steels had a good passivity in all structural states due to high content of chromium in solid solution, however at the stage of active corrosion the sharp acceleration of corrosion was observed after aging at 350 °C in investigated steels (Table 2, line 2).

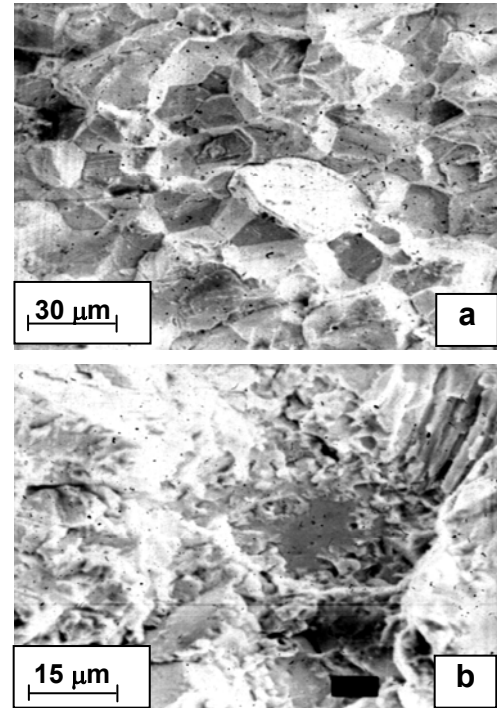


FIG. 1 FRACTURE SURFACE AFTER SCC OF CR18NI2N1STEEL SUBJECTED OF AGING AT 350 (a) AND 600 °C (b)

After quenching from 1200 °C an austenite and carbides with surface martensite were noted in carbon steel as well as in nitrogen steels (Fig. 2, a, b). The structure of high nitrogen steels varied with rising of aging temperature identically with some more stability of austenite in nickel-free steel. The microstructure of nitrogen steels aged at 350 °C was almost similar to quenched one (Fig. 2, c), but after aging at 600 °C it was rather different (Fig.2, d).

Martensites of two morphologies with different lattice parameters  $a_{\alpha T}=0.287$  nm and  $a_{\alpha\sigma}=0.291$  nm was discovered in steel Cr18Ni2N1. As it was shown earlier [15] it was the thin dispersive martensite ( $\alpha_T$ ) on the grain boundaries and annealing twins (Fig. 2, b, c) and lens-formed stress-induced martensite ( $\alpha_\sigma$ ) in the middle of grain (Fig.2, d), which formed respectively from austenite depleted and enriched with chromium and nitrogen. The same picture was found after SCC. The results of X-ray diffraction analyses are shown in Table 2.

TABLE II PHASE COMPOSITION AND CORROSION PROPERTIES OF THE Cr18Ni2N1 STEEL AFTER VARIOUS TREATMENT

№ of treatment		Scheme of treatment	Content of phases, %*						Km, g/m <sup>2</sup> .h	Δσ <sub>st</sub> , %
			In the initial state			At the surface of fracture after SCC				
			γ	α <sub>T</sub>	α <sub>σ</sub>	γ	α <sub>T</sub>	α <sub>σ</sub>		
Aging without CPD	1	Quenching from 1200 °C	95	5	0	62	26	12	2,0	35
	2	1200 °C + 350 °C, 2 h	95	5	0	58	22	20	30,0	37
	3	1200 °C + 400 °C	95	5	0	62	22	16	8,0	34
	4	1200 °C + 600 °C	50	44	6	28	64	8	1,0	29
	5	1200 °C + 700 °C	0	100	0	0	100	0	0,5	21
CPD before aging	6	1200 °C + 8% CPD + 350 °C, 2 h	89	5	6	-	-	-	10,0	-
	7	1200 °C + 12% CPD + 350 °C, 2 h	87	5	8	-	-	-	11,0	-
	8	1200 °C + 20% CPD + 350 °C, 2 h	50	15	35	40	15	45	55,0	42
	9	1200 °C + 24% CPD + 350 °C, 2 h	80	7	13	-	-	-	50,0	-
	10	1200 °C + 20% CPD	95	5	0	-	-	-	0,3	-
CPD after aging	11	1200 °C + 350 °C, 2 h + 20% CPD	95	5	0	95	5	0	2,5	10
	12	1200 °C + 400 °C + 20% CPD	95	5	0	-	-	-	0,3	-
	13	1200 °C + 600 °C + 20% CPD	50	50	0	-	-	-	0,8	-
	14	1200 °C + 700 °C + 20% CPD	0	100	0	-	-	-	0,4	-

\* Without excess phases.

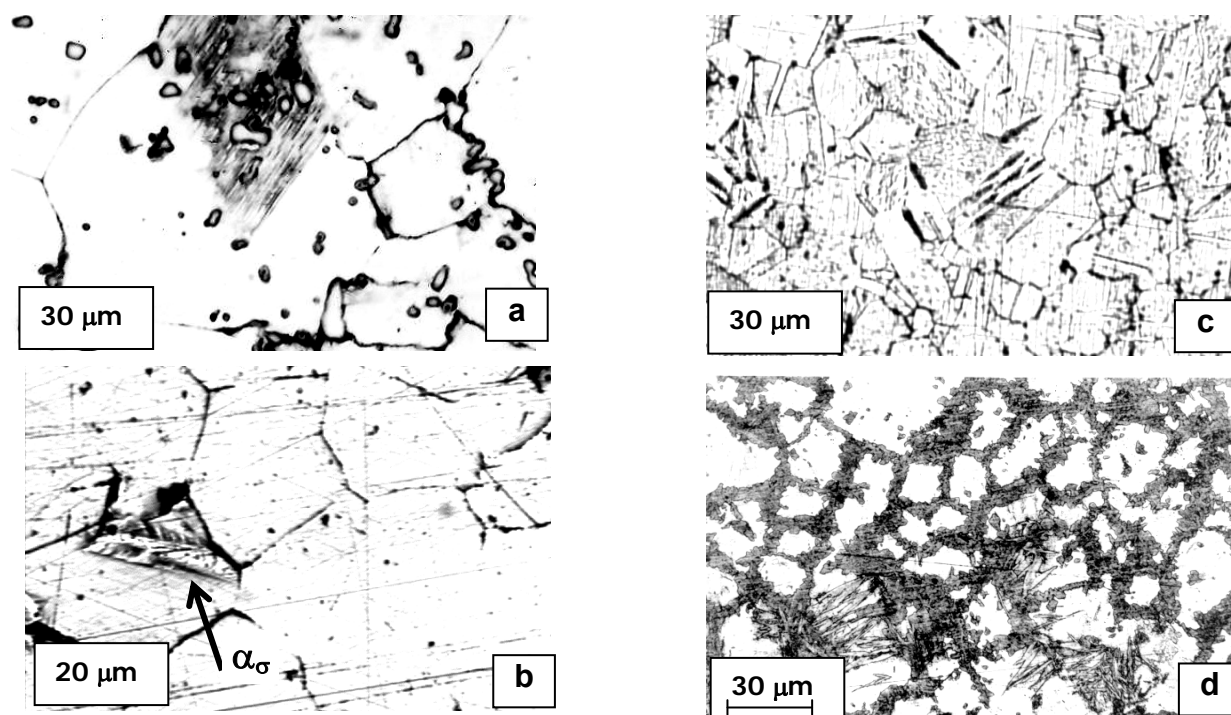


FIG. 2 MICROSTRUCTURE OF STEELS CR18C1 (A), CR21N1 (B) QUENCHED AND CR18Ni2N1(C, D) AGED AT 350 AND 600 °C

According to comparison of SCC tests and X-ray diffraction results the correlation between the value of relative strength reduction ( $\Delta\sigma/\sigma_k$ ) and quantity of stress-induced martensite, formed directly during the tests, is observed in high nitrogen steels. The biggest portion of martensite  $\alpha_\sigma$  in structure of steel was noted after aging at 350 °C.

By transmission electron microscopy of thin foils the evolution of structure in steel Cr18Ni2N1 under the aging was studied. The structure of quenching steel consisted of austenite (Fig. 3, a-c), rather big particles of nitrides  $\text{Cr}_2\text{N}$  retained after heating along the grain boundaries and not more than 5% of  $\alpha_\tau$ -phase as a result of martensitic transformation in depleted austenite near the nitrides (Fig. 3, d-f). The nitride  $\text{Cr}_2\text{N}$  has a hexagonal crystalline structure with lattice parameters  $a=0.4805$  nm,  $c=0.4480$  nm. The crystals of surface martensite formed at electrolytic polishing of foil could be seen as well (Fig. 3, g).

After aging at 350 and 400 °C besides  $\alpha$ - and  $\gamma$ -phase ultra dispersive nitrides CrN with FCC crystalline ordered structure and lattice parameter  $a_{\text{CrN}}=0.415$  nm formed from a supersaturated  $\gamma$ -solid solution due to homogeneous isomorphic decomposition were observed (Fig. 4, a-c). The crystals of martensite  $\alpha_\sigma$  were found as well. Both phases CrN and  $\alpha_\sigma$  were noticed nearby the extinction contours in the places of foil bending (Fig. 4, d-f). After 2-hour aging at 500 °C besides  $\alpha_\tau$ -phase in the settlement of discontinuous decomposition on the grain boundaries (Fig. 5, a-c) the single crystals of  $\alpha_\sigma$ -martensite were discovered (Fig. 5, d). At temperature 600 °C fresh portions of discontinuous decomposition settlements consisting of nitrides  $\text{Cr}_2\text{N}$  and  $\alpha_\tau$ -martensite was observed (Fig. 6).

The electric resistance of the steels Cr21N1, Cr18Ni2N1 and Cr18C1 has been measured depending on time of aging in isothermal conditions at 300, 400 and 500 °C (Fig. 7). These dependences were shown to be the same for high nitrogen and high carbon steels. At the initial stages of process (3-6 h) we can see a sharp electric resistance decrease which becomes bigger as the temperature of aging grows. The effect of aging depends on a super saturation degree by interstitial atoms of austenite quenched but at the same content of carbon and nitrogen (about 0.9%) it's displayed stronger in high nitrogen steels. With the increasing of the temperature at this stage of aging a difference between the curves reduced and they practically coincided at 500 °C. The stress recovery at the heating was responsible for decrease of

electric resistance of the steels (sharp decrease of  $\Delta R/R$  – pre-aging stage).

At a longer time of heating it was an increase of electric resistance most pronounced at 300 °C. This stage continued not more than 7 hours at 300 °C, 4 hours – at 400 °C and 1 hour – at 500 °C and associated with the separation of the solid solution with chromium (as well carbon in carbon steel) during these exposures because of the conductivity electrons are scattered by these enriched in chromium clusters [16] (sharp increase of  $\Delta R/R$  – stage 1).

Longer aging influenced on the curves character differently depending on the temperature of aging. It is necessary notice a stabilizing of decomposition of austenite in carbon steel at 300 and 400 °C and in nitrogen steels only at 300 °C. It is connected with the forming of carbides in carbon steel, but not completing of nitrides formation in nitrogen steels. It needs to continue the heating and further slow increasing of electric resistance which observed at 400 °C in these steels confirmed it. This process associated with the ordering in chromium enriched clusters with nitrogen that was able at an exposure at this temperature or under stresses (nearby the extinction contours – Fig. 4, a) if exposure was not enough. Thus the formation of carbides or nitrides characterized with unchanging or slow increasing of  $\Delta R/R$  – stage 2.

Decreasing of electric resistance at 500 °C has been shown in all investigated steels and was connected with dissolving of clusters enriched in chromium (slow decreasing of  $\Delta R/R$  accordingly in carbon and nitrogen steels – stage 3).

Effect of cold plastic deformation on the phase content and corrosion properties of the steel Cr18Ni2N1 is presented in Table 2. CPD at an 8-24% reduction was combined with aging at 350 °C by two ways: before (lines 6-10 in Table 2) and after aging (lines 11-14) as compared to the undeformed state (lines 1-5). It follows from Tab. 2 that CPD at 8-12% reduction before aging at 350 °C decreases to thrice a corrosion rate in spite of some increasing of  $\alpha_\sigma$ -martensite content. On the contrary CPD at 20% reduction increases nearly twice the corrosion rate because of considerable depleting of austenite and as consequence increasing of  $\alpha_\tau$ -martensite content after strain aging. Furthermore this treatment condition negatively influenced on the SCC of this nitrogen steel and increased of  $\alpha_\sigma$ -martensite content if to compare with undeformed state (lines 2 and 8). It is known [17] that a low level of stresses ( $\sigma < \sigma_{tr}$  – threshold stress)

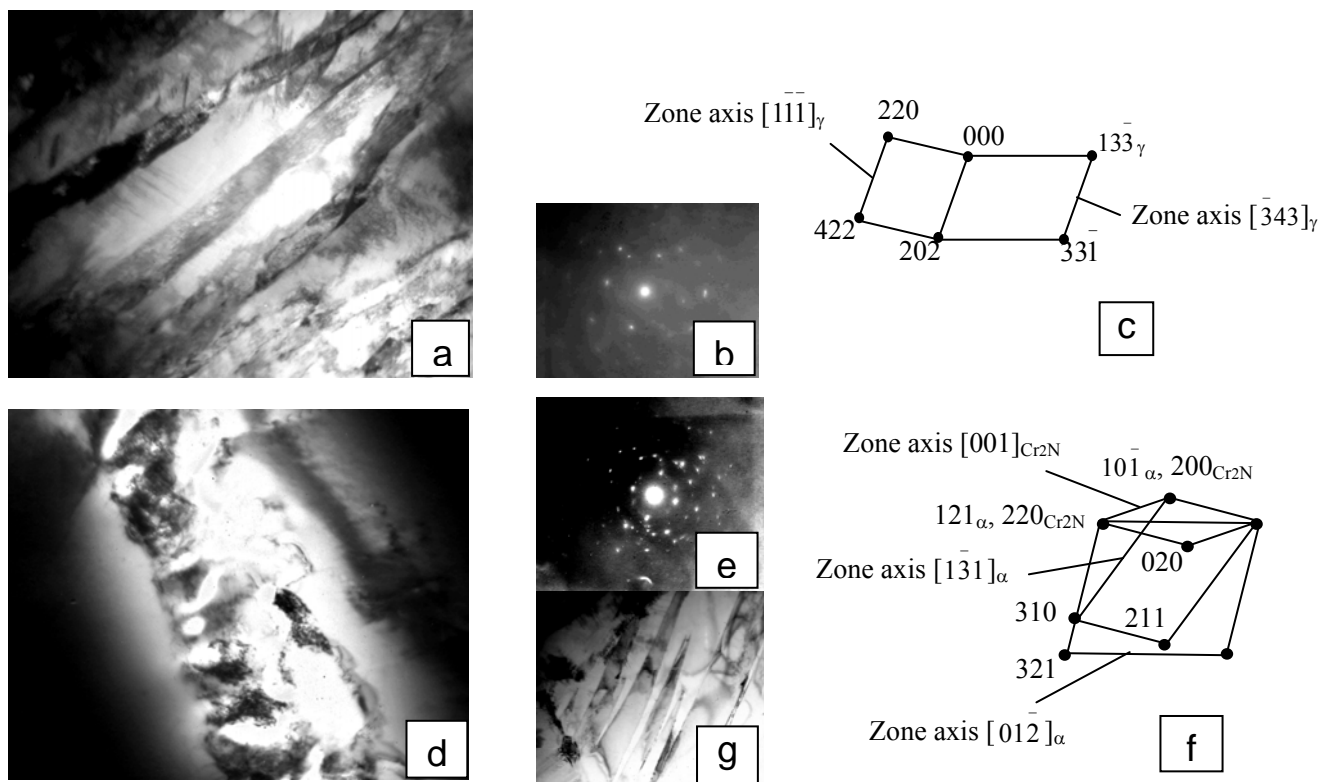


FIG. 3 STRUCTURE OF QUENCHED STEEL CR18NI2N1: A – SUB GRAIN STRUCTURE OF AUSTENITE; B – SELECTED-AREA ELECTRON DIFFRACTION PATTERN TAKEN FROM REGION (A); C – KEY PATTERN; D – STRUCTURE OF GRAIN BOUNDARY; E – SELECTED-AREA ELECTRON DIFFRACTION PATTERN TAKEN FROM REGION (D); F – KEY PATTERN; G – SURFACE MARTENSITE

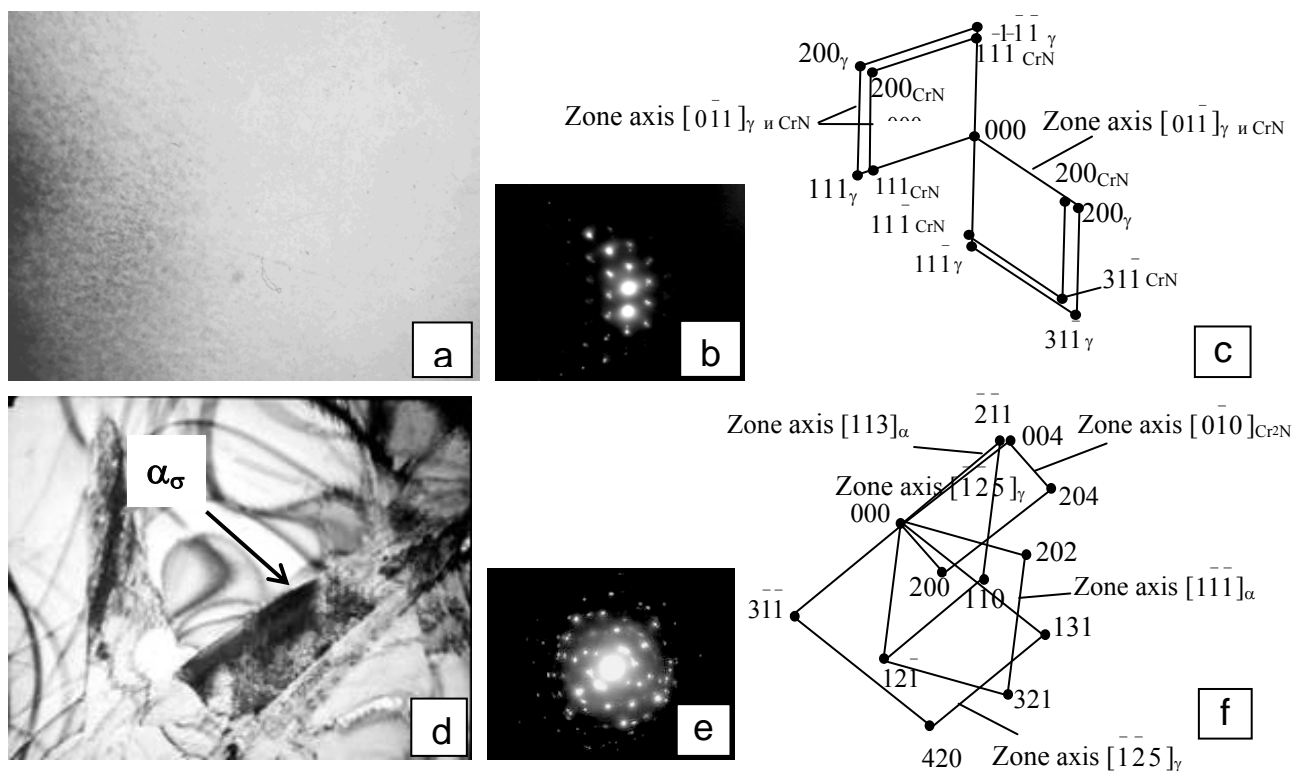


FIG. 4. STRUCTURE OF AGED AT 350 °C STEEL CR18NI2N1: A – HOMOGENEOUS DECOMPOSITION OF AUSTENITE; B – SELECTED-AREA ELECTRON DIFFRACTION PATTERN TAKEN FROM REGION (D); C – KEY PATTERN; D – EXTINCTION CONTOURS AND MARTENSITE  $\alpha_\sigma$ ; E – SELECTED-AREA ELECTRON DIFFRACTION PATTERN TAKEN FROM REGION (D); F – KEY PATTERN

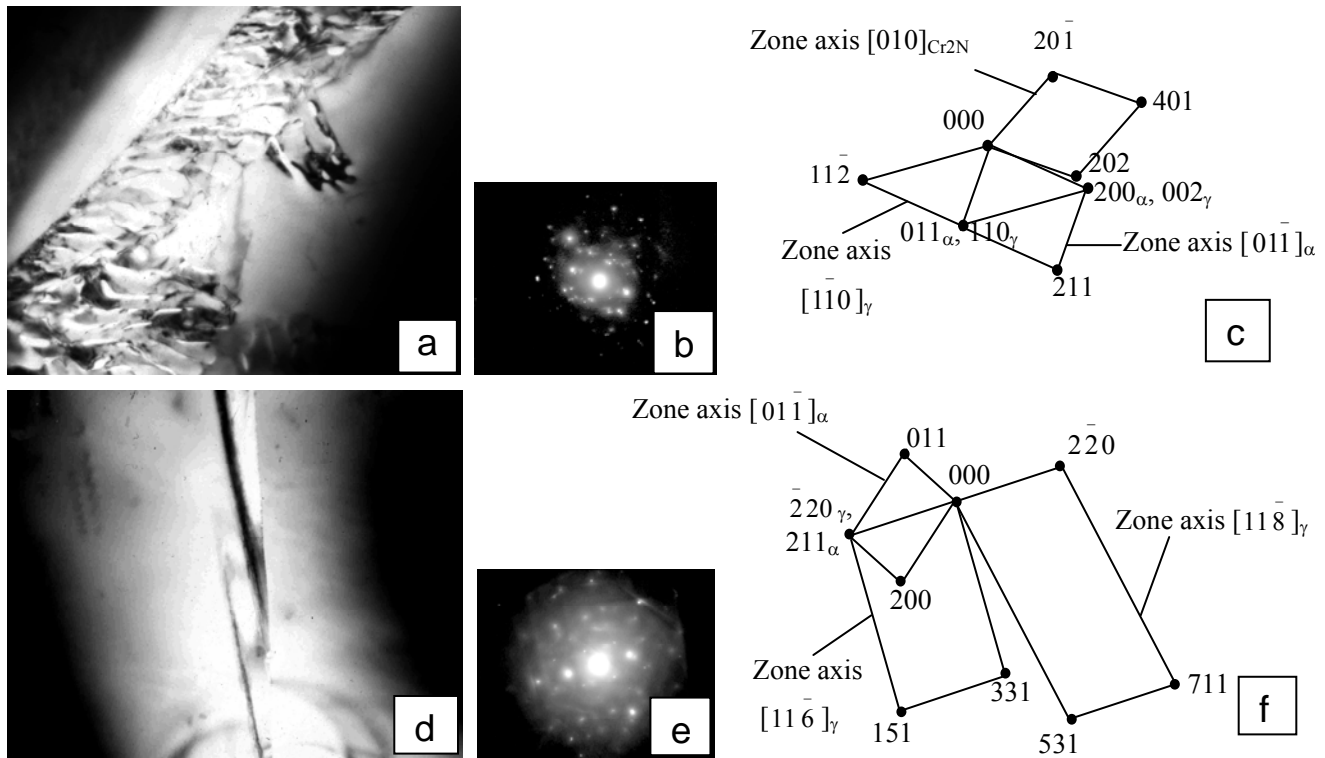


FIG. 5 STRUCTURE OF AGED AT 500 °C STEEL CR18NI2N1: A – GRAIN BOUNDARY; B – SELECTED-AREA ELECTRON DIFFRACTION PATTERN TAKEN FROM REGION (A); C – KEY PATTERN; D – MARTENSITE  $\alpha_{\sigma}$ ; E – SELECTED-AREA ELECTRON DIFFRACTION PATTERN TAKEN FROM REGION (D); F – KEY PATTERN

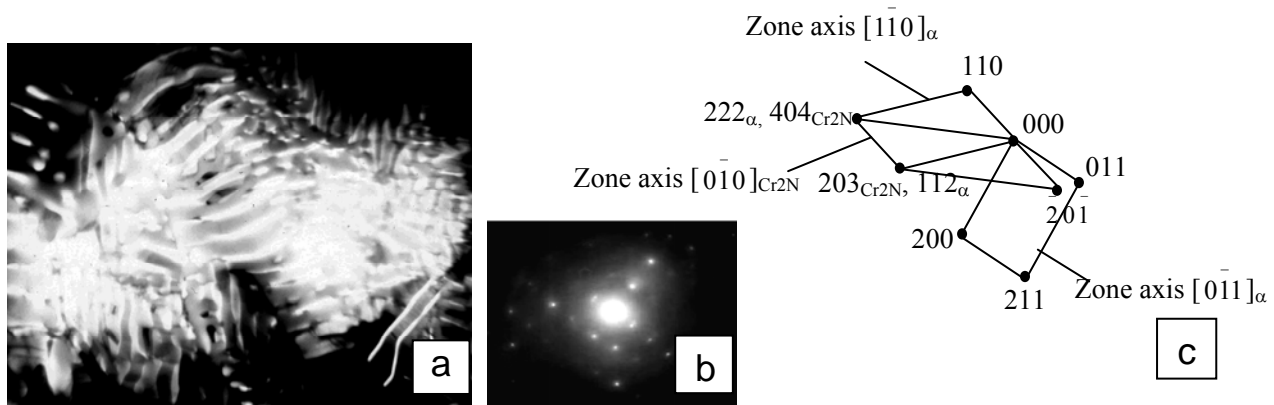


FIG. 6 STRUCTURE OF AGED AT 600 °C STEEL CR18NI2N1: A – GRAIN BOUNDARY; B – SELECTED-AREA ELECTRON DIFFRACTION PATTERN TAKEN FROM REGION (A); C – KEY PATTERN

does not considerably increase the corrosion current at SCC of iron but a high level of them ( $\sigma > \sigma_{tr}$ ) causes a significant growth of this one (Fig. 8). And taking into consideration the same to delayed failure [18, 19] mechanism of SCC, when the main depolarization process is hydrogen ions ( $H^+$ ) discharge on the ordered clusters to atoms (H), they become the effective sub micro cathodes in electro-chemical process on the surface of the steel. The atoms of hydrogen can join to molecules ( $H_2$ ) significantly

increasing their size and level of stresses in the clusters. That is why the stress-martensite can be formed from those local areas of structure.

After 24% reduction the austenite has more stability than after 20% reduction because of strain recovery, that's confirmed by dependence of micro hardness from deformation degree (Fig. 9). Fig. 10 shows the structure of deformed before aging at 350 °C steel with the electron diffraction pattern where  $\alpha_{\sigma}$ -martensite can be noted.

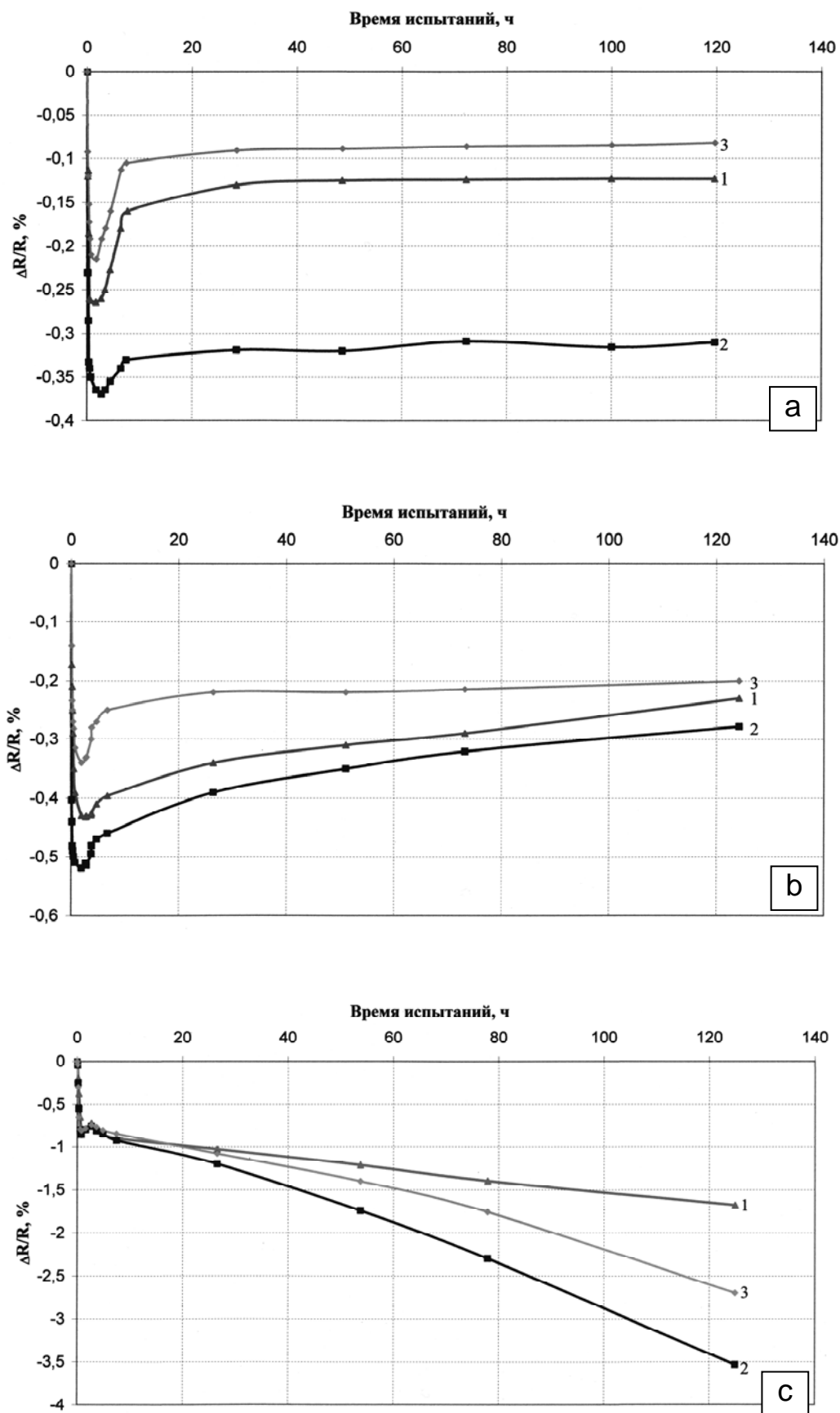


FIG. 7. KINETICS OF ELECTRIC RESISTANCE CHANGE IN INVESTIGATED STEELS CR21N1 (1), CR18Ni2N1 (2) И CR18C1 (3) DURING THE AGING AT: A – 300 °C; B – 400 °C; C – 500 °C

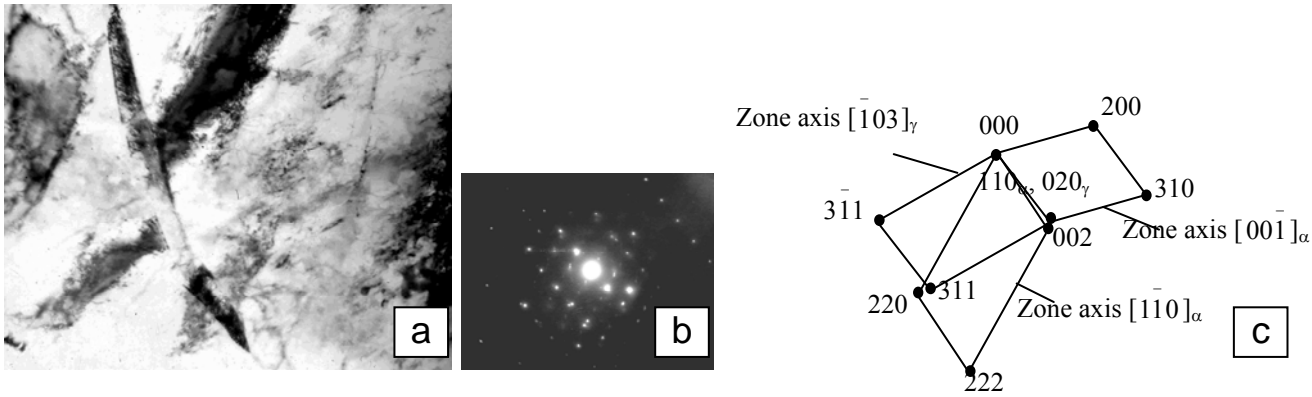


FIG. 10 STRUCTURE OF DEFORMED AT AN 8% REDUCTION BEFORE AGING AT 350 °C STEEL CR18Ni2N1: A – MARTENSITE  $\alpha_M$ ; B – SELECTED-AREA ELECTRON DIFFRACTION PATTERN TAKEN FROM REGION (A); C – KEY PATTERN

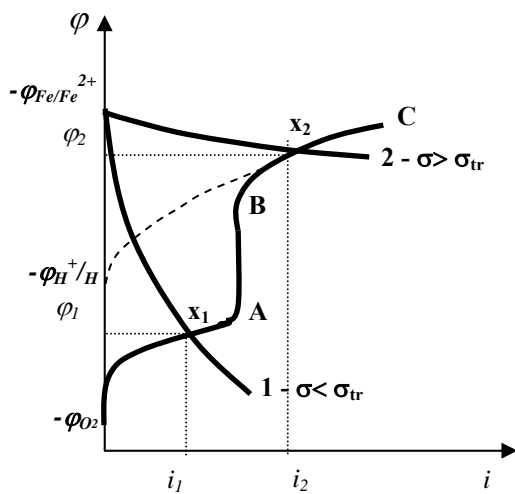


FIG. 8 POLARIZATION DIAGRAM OF CORROSION AT SCC:  $\phi_{O_2}ABC$  – CATHODE CURVE;  $\phi_{Fe/Fe^{2+}}X_1$  AND  $\phi_{Fe/Fe^{2+}}X_2$  – ANODE CURVES AT A DIFFERENT STRESS

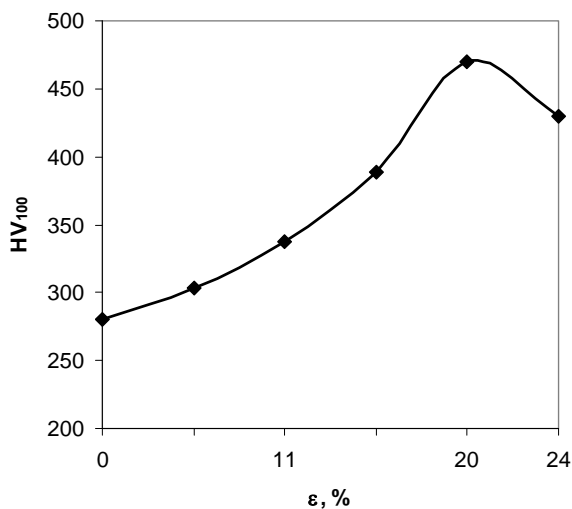


FIG. 9 DEPENDENCE OF THE MICRO HARDNESS ON THE DEGREE OF CPD PRECEDED THE AGING AT 350 °C IN STEEL CR18Ni2N1

Treatment with CPD at 20% reduction after aging at 350 °C is found to be more effective: the corrosion rate decreased by an order and the reduction of strength at SCC was about a quarter of magnitudes of undeformed state. This positive influence of CPD is probably connected with breaking the order in metastable clusters which could be acceptors of discharged hydrogen and nucleus of stress-induced martensite at SCC.

### Conclusion

The structure of quenched steels Cr21Ni1, Cr18Ni2N1 and Cr18C1 with high content of nitrogen and carbon, respectively 1.021, 0.899 and 0.920% consists of metastable austenite with nitrides or carbides settled down the grain boundaries. Also a small content of  $\alpha$ -phase takes place near the nitrides/carbides or as a result of polishing.

Two stages of decomposition of austenite at tempering were shown to be in nitrogen steels by electric resistance: separation of the solid solution with chromium followed by ordering of these clusters with nitrogen which induced by time-exposure or stresses.

The highest hardness, corrosion rate and stress corrosion cracking susceptibility in HNS were observed at the second stage of aging. This structural state characterized of ordered enriched with chromium and nitrogen clusters which had an ability to transform into a stress induced martensite.

Cold plastic deformation at 8-12% reduction preceding the aging at 350 °C is shown to decrease three times the corrosion rate of the steel Cr18Ni2N1 as compared to the undeformed state. The role of CPD at this degree consists of accelerating of nitrides CrN precipitation without intermediate stage of ordering



clusters. A thermoplastic treatment by 20% CPD after aging at 350 °C is more effective because of destroying already formed ordered clusters, which are the main reason of high susceptibility to SCC.

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